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Research highlights: The determination and origin of fibre clogging in membrane bioreactors

- First study of membrane channel clogging in membrane bioreactors (MBRs) by coarse suspended solids (CSS) based on empirical measurement of 10-12 full-scale MBR plants
- New on-site methodology developed for assessing “ragging” propensity: the agglomeration of fine fibres to form long “rags” in the MBR tanks.
- Behaviour of washed CSS filtered from the MBR sludge using a 4 mm – aperture net found to be identical to that of cotton wool fibres.
- Correlation between filterability and the concentration of CSS closer than that between filterability and classical mixed liquor suspended solids (MLSS) concentration.
- Heuristic observations revealed plants using macerating screens to be more prone to clogging than those using classical screen removal, consistent with the supernatant from the macerators returned to the process containing the fine cotton wool fibres responsible for ragging.

The determination and origin of fibre clogging in membrane bioreactors

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Abstract

Membrane channel clogging in membrane bioreactors (MBRs) has been largely unexplored by the research community, despite it being widely recognised as the main impediment to sustainable MBR operation by practitioners, and reflects the difficulty of examining clogging with the same scientific rigour as membrane fouling.

The incidence of clogging/"ragging" has been assessed across 10-12 full-scale flat sheet immersed MBRs, equipped with inlet screens of similar rating, and physical characteristics of the sludge examined. Measured sludge quality determinants included classical mixed liquor suspended solids (MLSS) concentration and filterability measurement, as well as a series of novel sludge characterisations based on determination of coarse suspended solids (CSS) concentration using sieves or nets of aperture sizes between 150 and 4000 μm. A further rudimentary test was conducted on the coarse filtered solids to assess the mechanical integrity of the agglomerated material.

Results showed there to be a reasonable correlation between filterability and concentration of super-4 mm particles, whereas no such correlation existed between filterability and MLSS. This indicated the nature of the solids, and particularly the highly-coarse particulate content, to be critical in determining filtration resistance. Both filterability and CSS concentration also correlated with heuristically-assessed clogging propensity, with those plants fitted with macerator screens being more prone to clogging than those with more conventional screens where screenings were removed entirely.

The coarse suspended solids were found to readily agglomerate to form characteristic "rags", i.e. 10-15 cm-long braids of aggregated material, which were self-supporting when suspended in air. The mechanical integrity of these fibres was unaffected by chemical treatment either with detergent or hypochlorite, indicating a degree of robustness of the rags which were formed primarily of cellulosic matter. Identical properties were recorded from experiments conducted on cotton wool filaments, suggesting ragging to relate to disposal of cotton wool-based products to sewer.

Keywords *membrane bioreactor, clogging, ragging, sludge, flat sheet, cellulose*

1 Introduction

Limited peer-reviewed literature exists describing comparative operational experience of large full-scale municipal immersed membrane bioreactor (iMBR) installations, although two recent reference texts are available [1-2]. Reports have tended to be either case studies based on a one or two installations [3-4], or else have been surveys of fouling propensity with respect to foulant species [5-6]. Despite significant emphasis on membrane fouling in published scientific papers on MBRs [7], a recent survey revealed 41% of iMBR practitioners to identify screening and clogging as main impediments to sustainable iMBR operation [8] - topics featuring only in a few reports [9-15].

Clogging contributes to permeability decline through the agglomeration of solids within or at the entrance to the membrane channels. There are two recognisable clogging behaviours: *sludging*, or the filling of membrane channels with sludge solids, and *ragging* or *braiding,* the blocking of membrane channels with particles agglomerated as long rag-like particles [1]. Very few studies of iMBR clogging exist, one example being the study of membrane aeration rate on clogging within fibre bundles [11], and no published studies of ragging are evident. Sludging depends on plant operational flux and flux distribution, influent screen type and rating, membrane aeration distribution, plant control and membrane module design. It is understood that ragging is more relevant to municipal plants, since the rags are primarily made up of cellulosic fibres and hairs [10, 12-15]. It must also be assumed that both processes are dependent on concentration and characteristics of the particles within iMBR system mixed liquors, and thus the sludge quality, but do not necessarily relate directly to widely studied fouling constituents.

The sludge quality strongly depends on the feed sewage characteristics, and it has been assumed by most practitioners that the clogging propensity relates to the level of pretreatment applied. Specifically, the solid matter contributing to clogging is assumed to pertain to the type, aperture size and geometry, and cleaning mechanism of the fine screen [1, 10, 12-15], and some studies of solids removal by sewage screening systems have been provided [12-15]. Mason *et al* [10], following the study of operational characteristics of three full scale municipal flat sheet iMBR plants, recommended use of inlet screens with high capture efficiency for lint and hair and described successful installation of *Copasac* nets to reduce lint build up within the system. Frechen and co-workers [12-13] reported work on the development of a suitable method to describe braid formation capacity, surmising that a threshold concentration of hair and fibres is required for rag/braid formation. The authors reported a linear relationship between the weight of rag/braid formed and the screened sewage suspended solids concentration, with the gradient representing ragging propensity. These authors found that the size fraction of feed sewage below 190 μm not to contribute to ragging. However, none of these studies have considered in-situ agglomeration of solids, the actual process of rag formation, or the impact of the physical sludge constituents.

Cellulose fibres are a common ingredient of bathroom products, able to form water absorbent yarns having some tensile strength. Yarn strength is built upon the friction force between bound individual fibres [16] and can be increased by wetting [17]. The most commonly used cotton cellulose fibre has typically 12 to 25μm diameter and 1.5 to 5.5cm length [18]. Cellulose is natural, polar (hydrophilic), insoluble polymer which is highly resistant to biodegradation [19-20]. Even at the relatively long sludge ages typically applied to MBR processes [1] only partial cellulose decomposition arises [19-20], such that the fibres are largely retained in the system. It has, however, been noted by researchers analysing cellulases (cellulose-hydrolysing enzymes) at the molecular level that many cellulases may modify the cellulose fibre surface, as well as releasing small particles consisting of bundles of microfibrils [21].

A number of studies exist where researchers have used sludge filterability measurement as one of the main iMBR sludge quality indicator [22-24]. The leading flat sheet (FS) membrane manufacturer recommends specific filterability ranges for iMBR operation where filterability in this case is defined as filtrate volume per specific time period (ml/5min) under a defined set of conditions [25-26]. In determining in-situ filterability results with concentration and size of the MBR mixed liquors floc solids, Lousada-Ferreira et al [22] showed filterability, defined as resistance increase following permeate production (ΔR_{20}), to be unrelated to particle concentration within the 10-100 μm size range and the normal range of mixed liquor suspended solids (MLSS) concentration. Against this, the particle concentration over the 0.4-5 μm size range correlated reasonably well (R^2=0.7) with filterability as well as the ΔR_{20} value [24], presumably reflecting membrane pore plugging by colloidal matter.

In the current study iMBR plants sludge characteristics, and specifically their clogging/ragging propensity, were correlated with the filterability and particle size fraction using simple practical tests, encompassing a wide range of particle sizes (Fig. 1) which could ultimately be performed primarily on site. The study aimed to determine possible impact of pretreatment on clogging propensity, and specifically whether clogging is influenced by the type of screenings handling system installed. Tests range from the standard manufacturer's recommended filterability test, based on 2.5μm pore size filter paper, to sieving using standard sieves or nets of different apertures to measure the coarse particle concentration in the mixed liquor. In doing so, it was intended to advance the understanding of the iMBR plant clogging phenomena through identifying (a) the aperture sieve or net size most apposite for clogging propensity determination, and (b) the most appropriate screenings management.

2 Materials and Method

2.1 Population and plants grouping

Twelve iMBR plants were selected for sampling. To limit variability arising from catchment and operating conditions, all plants chosen were located within same region, were fed with 90-100% municipal wastewater, and operated by the same water company. All plants were equipped with same type of *Kubota* FS membranes in single or double deck configuration, shared a similar overall design, and were operated with similar setpoints for between 3 and 6 years post commissioning. For two of the 12 plants only the plant characteristics were available: no sludge sampling took place at these sites.

All plants selected were equipped with two-stage inlet screening: two-dimensional (2D) 6mm followed by 2D 3mm, with screens of similar screenings capture efficiencies according to a previous evaluation report [15]. Two thirds of the plants were installed with macerating screenings handling (MSH) units on one or both screening stages. The MSH system degrades captured screenings to 10 mm particle size before suspending and transferring them to a perforated bowl separator (PBS) unit. The PBS produces a decanted liquid stream containing suspended matter returned upstream of the screen, the coarse solids being disposed of. MSH units thus typically generate reduced volumes of screenings compared to the more conventional screw conveyor, compacting and dewatering units installed at the screening stages of the remaining four plants. The plants could thus be divided into two groups:

macerating screenings handling (MSH, 8 plants) and non-macerating screenings handling (nMSH, 4 plants). The two plants for which no sludge sampling took place were both MSH plants.

Anecdotal information on plant clogging propensity was acquired through interviewing the senior process advisor for the plants who had extensive knowledge of the behavioural characteristics of all twelve plants throughout their period of operation. Consequently, the plants were segregated on the basis of their behaviour into two groups: clogging resistant (6 plants) and clogging susceptible (6 plants).

2.2 Applied tests

Test methodologies employed for solids concentration measurement and characterisation are presented in Table 1 and illustrated in Figure 1. The Standard Method No. 2540 D was used to measure mixed liquor suspended solids (MLSS) concentration. Filterability and solids concentration determination methods comprised the manufacturer's recommended mixed liquor filterability test [25-26], Table 1, the manufacturer's proposed coarse suspended solids (CSS A and B) measurement, and three novel CSS determine concentrations of particles above 150, 500 and 4000 µm, including free floating rags/braids. Finally, a novel analysis of the actual retained rags was undertaken (CSSR) to determine solids properties.

Figure 1. Applied tests retained material particle size compared with inlet screens installed.

A single set of mixed liquor/sludge samples was collected from ten of the twelve sites and analysed in the laboratory, with the CSS4000 analysis performed on site. Samples were taken during three days of site visits and samples stored at <5 Cº prior to laboratory analyses. Statistical analyses were performed to establish significant differences in data sets. Tests included coefficient of variation, single-sided T-test with unequal variances, Kruskal-Wallis one-way analysis of variance by ranks, and linear regression analysis.

3 Results and Discussion

3.1 Development of CSS test methodology.

Tests measuring coarse suspended solids were developed prior to the main research programme. Table 2 presents results for the coefficient of variation for five test procedures: Filterability, and the CSS-A, CSS150 (modified CSS-A procedure) CSS500 and CSS4000 tests. According to these data, the CSS150 and CSS500 tests appeared to be the most reproducible with coefficient of variation values for three repetitions below 10%. To compensate for the higher filterability and CSS4000 coefficients of variation these two tests were triplicated in the trials.

Table 1: Applied methodologies

Table 2: Coefficient of variation values, three test repetitions

3.2 Statistical analysis of data from plant families

All four nMSH plants were also classed as clogging resistant and six of the eight MSH plants classed as clogging susceptible. This would appear to indicate that returning the supernatant from the macerated screenings to the head of works promotes clogging.

Statistical analysis of the sludge characterisation data for the ten plants from which sludge samples were taken comprised a one-sided T-test with unequal variances and Kruskal-Wallis (K-W) test (Tables 3-4). The K-W test (a non-parametric version of the T-test) was used since, in some cases, the variances within each group were very different, which can cause the T-test to show misleading results. For both statistical tests the aim is to demonstrate a statistically significant difference in the two data sets (MSH vs nMSH, Table 3, and clogging susceptible vs. clogging resistant, Table 4). Confidence in any noted difference is represented by the p value, where by a value of <0.05 indicates $>95\%$ confidence.

Table 3: Results for the one sided T-test with Kruskal-Wallis analysis for macerating and non-macerating screenings handling plants.

Table 4: Results for the one sided T-test with Kruskal-Wallis analysis for clogging resistant and susceptible plants.

According to the data, the MLSS (or overall suspended solids) concentration, where high values are most often assumed to promote clogging [1], appears to have limited influence on clogging propensity, (T-test p value of 0.09, Table 4). There is an even lower significant difference ($p < 0.15$) in the two families of plants using macerating and non-macerating screenings handling systems (Table 3). The KW test data support this trend more emphatically (0.25 and 0.29 respectively).

For both of the two sets of families, more significant differences are evident for the other five physical sludge quality determinants. Sludge filterability was significantly lower, with very high confidence ($p<0.002$), for those plants fitted with MSH systems. Moreover, for all CSS determinants other than CSS-B, data indicate significantly greater CSS concentration values for the MSH-based plants (Table 3), with consistent trends across the clogging susceptible vs. clogging resistant plants. The mean filterability of clogging resistant plants was found to be higher compared to clogging susceptible plants at a high level of confidence ($p<0.02$, Table 4), inferring that both the filterability test and CSS measurements – including the simplest on-site CSS4000 test ($p<0.05$) – provide an adequate indicator of clogging propensity. The same conclusions can be drawn from the K-W test data.

3.3 Data correlations

Data revealed filterability to change by almost an order of magnitude within MLSS range of 11-19g/l, but with no apparent consistency. Operation of all nMSH plants was within the ranges of MLSS concentration (10-20 g/L) and filterability (>10ml/5min) recommended by the manufacturer [25-26]. Only two MSH, clogging-susceptible plants, demonstrated poor filterability (<5ml/5min), with data for the remaining four plants falling between 5 and 10 ml/5min. Regression analysis indicated no clear trend between either filterability or CSS concentration with MLSS concentration (Table 5), consistent with some previous reports [22].

Table 5: Relationships observed between MLSS, Filterability and CSS tests data.

Against this, the CSS500 and CSS4000 data displayed a reasonable linear correlation with filterability, with the strongest correlation observed for the CSS4000 data (Fig. 2). The CSS4000 and CSS500 data also appear to be inter-related (Fig. 3), as would be expected. However, no correlation with filterability was noticeable for the smaller coarse particle sizes (CSS-B and CSS150), corroborating previously reported findings for resistance which was found to be unrelated to particle concentration within the 10-100μm size range [21]. It was further observed that at lower levels of filterability it was more difficult to dewater the filter cake from the CSS4000 test, possibly demonstrating its relative hydrophilicity. One of the potential explanations for this relationship may be the presence of cellulolytic microorganisms: strongly adsorbed cellulases may have adversely affected dewatering of CSS4000 captured material - with simultaneous release of small particles, bundles of microfibrils, by CBD (cellulose binding domains) activity - affecting filterability by promoting levels of 0.4-5μm particles [24].

Figure 2. Filterability vs. CSS4000 for different screenings handling systems

Figure 3. Relationship between CSS500 and CSS4000 results grouped for clogging propensity.

3.4 Solids analysis

Low quantities of >20 mm-long hairs and fibres were observed in the material retained by the CSS150-500 tests (Fig. 4). Substantial material from the sampled sludge was retained with the 4 mm-aperture net (CSS4000), notwithstanding the 3 mm rated screen upstream. This indicates a degree of agglomeration of the particles, which were not evident in the other sieved solids where the sludge volume sampled was three orders of magnitude less than that of the CSS4000 test. The material retained from the CSS4000 test from the two sites having the highest CSS4000 measurement was subjected to the empirical CSSR test. For both sites and all four fractions (a-d), rags were successfully reconstituted to form self-supporting rags when suspended in air (Fig. 5). Rags retained their mechanical integrity after washing (Fig. 5b), treating with surfactant (Fig. 5c) and after treating with strong oxidant (Fig. 5d). The results demonstrate the mechanical integrity of the rag/braid to be unaffected by surface activity, dissolved organic matter or hairs – the latter being readily disintegrated by strong hypochlorite solutions. Clearly, rags formed in MBRs relate largely to cellulosic materials.

Figure 4. Retained material from tests: CSS150 (left), CSS500 (centre), CSS4000 (right) for nMSH (top) and MSH (bottom) plants.

Figure 5. CSSR(a-d) test.

The origin of these rag-like cellulosic materials is of key interest. A rudimentary supplementary test was conducted whereby samples of bathroom tissue, paper towels and cotton wool were each suspended in water by chopping into lengths of <3 mm and blending suspensions in water in a high-shear mixer for 15s at a concentration of 200 mg/L before transferring to a 2L cylindrical container. It was found that, whereas the paper-based products could be suspended, the cotton wool formed large aggregates that could not be dispersed by mixing. On removing these aggregates with tweezers, rags identical in appearance to those from the CSSR(d) test (Fig 5d) were formed. No such rags were readily formed with the paper-based products.

4 Conclusions

Twelve full scale municipal iMBR plants, similar in design and operation, have been assessed in terms of clogging propensity, ten of which have been further sampled. Filterability, MLSS, and novel coarse suspended solids (CSS) determinations have been applied. Plants, selected based on their similarity in size and type of the fine screen, have been grouped as (a) clogging resistant and susceptible, and (b) equipped with macerating and non-macerating screenings handling system.

Statistical analysis of sludge data from ten of the plants revealed the physical screenings handling protocol, and specifically their maceration and return of the liquid fraction to the head of works, to impact manifestly on sludge quality. Sludge filterability and coarse particle concentration were found to be good indicators of clogging/ragging propensity, with filterability decreasing weakly exponentially (exponent coefficient = ~-0.004) with increasing concentration of super-4 mm particulate matter. MLSS concentration, on the other hand, was found to be unrelated to clogging propensity. The three novel CSS tests applied could potentially be used as sludge clogging propensity indicators, with the CSS4000 test being the simplest and correlating the most closely to filterability. CSS data at lower sieve apertures did not correlate with filterability, which would appear to be counter-intuitive given that filterability relates to plugging of an effective pore size of 2.5 μm. It is postulated that the observed trends may relate to the presence of the cellulolytic microorganisms and/or partly-biodegraded (hydrolysed) cellulose fibres, causing the release of microfibrils in the colloidal size range.

Rag reconstitution is a qualitatively determinable phenomenon. It was observed that a cellulose-based material comprising relatively short lengths of agglomerated fibres was captured by a sieve having 4 mm apertures. The captured material was successfully reconstituted into rags following washing, defragmenting, soaking in detergent, and soaking in a strong oxidant. Dispersed sub-3 mm lengths of cotton wool fibre were found to behave in identical fashion, suggesting the rags originate from cotton wool.

Results suggest that the simple on-site determination of coarse particle concentration is likely, at the very least, to provide a useful adjunct to recently proposed in-situ fouling determination [23, 27]. Rags appear to derive from cotton wool, indicating cultural/domestic habits to exert a strong influence on municipal wastewater treatability by MBRs: in the UK 57% of respondents to a survey disposed of foreign items to the domestic sewer at least once in single year, with 60% of the items being cotton wool based [28]. Clearly, however, more work is needed to allow ragging to be studied under more controlled conditions to identify its root causes and correlate it with feedwater quality, and so permit its more accurate predictive determination than that afforded in the current study.

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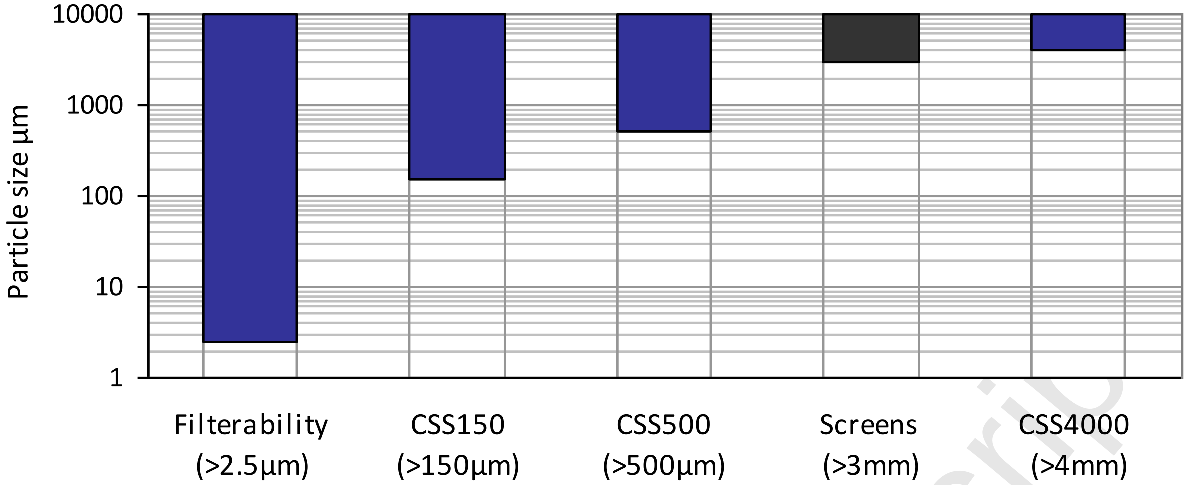
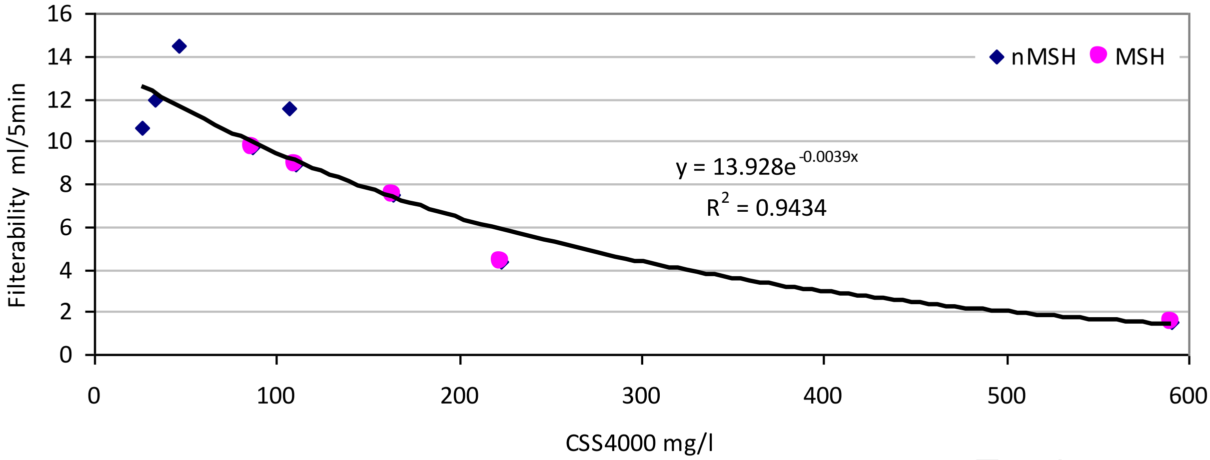
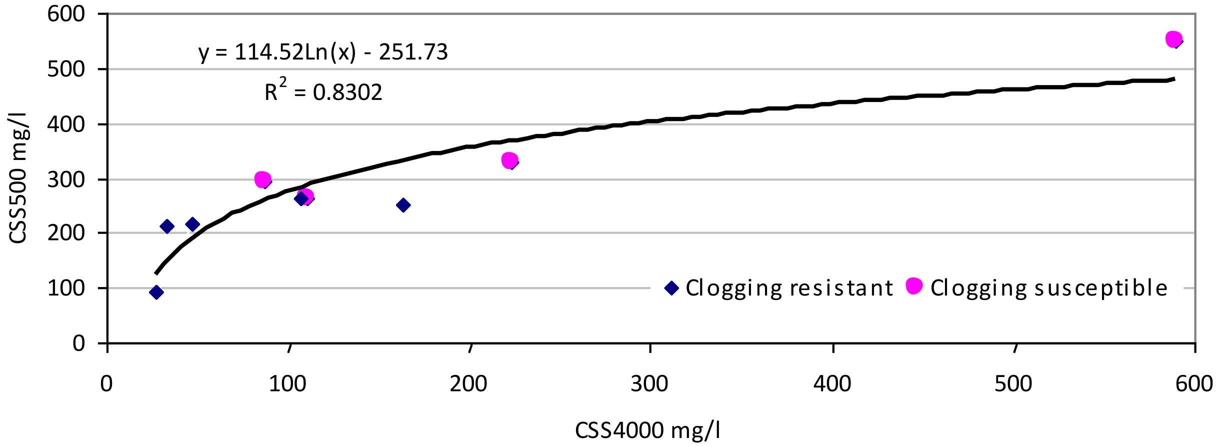


1 Applied tests for retained material particle size compared with inlet screens installed.

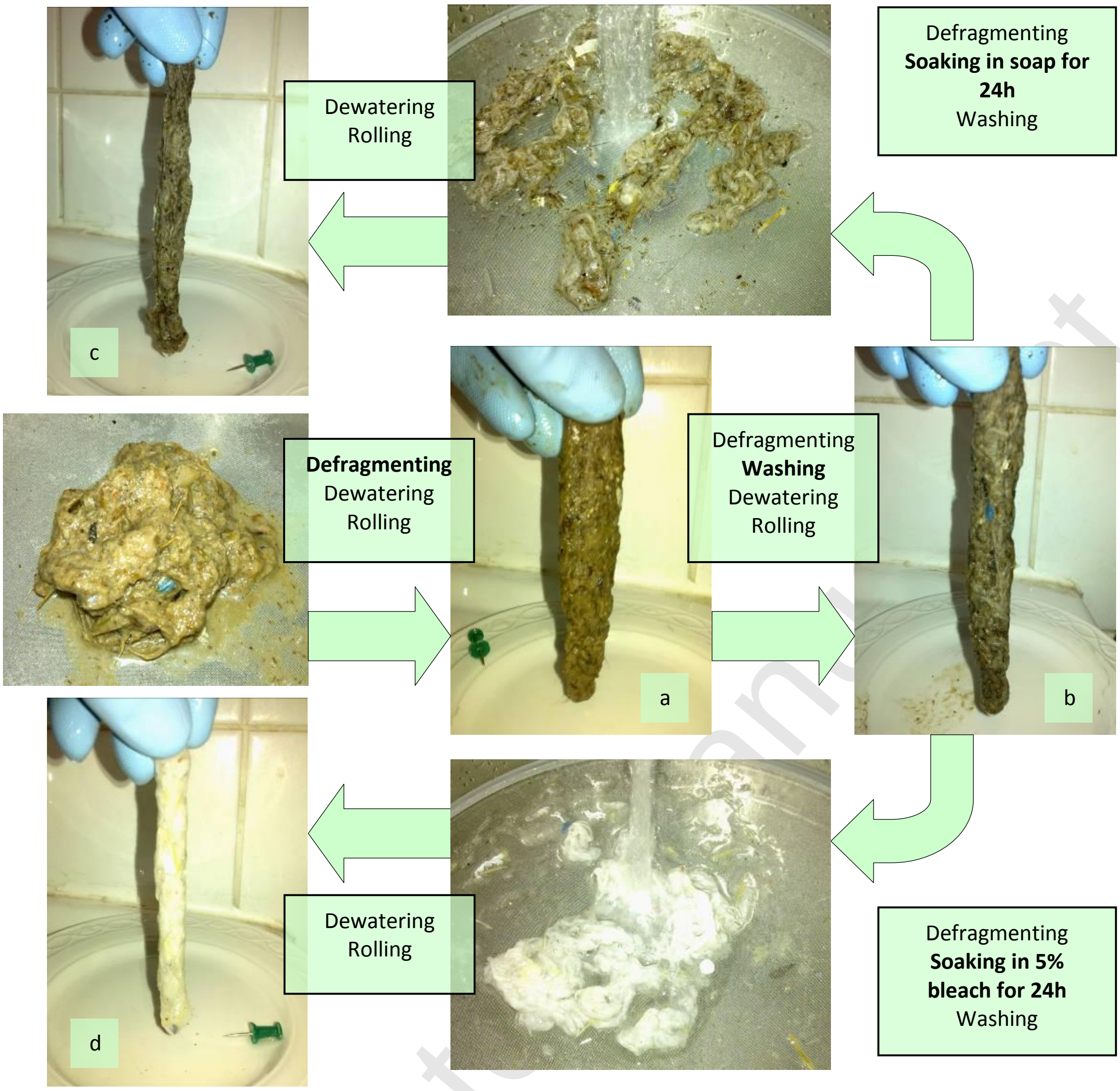


2 Filterability vs. CSS4000 for different screenings handling systems



3 Relationship between CSS500 and CSS4000 results grouped for clogging propensity.

4 Retained material from tests: CSS150 (left), CSS500 (centre), CSS4000 (right) for nMSH (top) and MSH (bottom) plants.



5 CSSR(a-d) test.

Table 1: Applied methodologies

Name	*Description*
MLSS	Industry standard mixed liquors test for total suspended solids: Standard Method No. 2540 D (Standard Methods Committee, 1997)
Filterability	Methodology proposed by membrane manufacturer. Folded 2.5 µm filter paper (Whatman, ashless, grade 42) used to filter 0.05 L of mixed liquor under gravity and total filtrate volume recorded after 5 minutes of filtration. Results expressed as filtrate volume after 5 minutes of filtering.
CSS-A	Coarse suspended solids measurement with methodology proposed by membrane manufacturer. 0.25 L sample of mixed liquor passed through 150 µm sieve for 2 minutes with gentle rinsing using tap water to assist sieving and isolate filter solids; remaining material collected and dried at 105 °C for 24h, and dried sample weighed. Results expressed as dry weight per litre.
CSS-B	Coarse suspended solids measurement modified from membrane manufacturer's methodology. 0.25 L sample of mixed liquor, passed through 150 µm sieve for 15 minutes with gentle rinsing using tap water to assist sieving and isolate filter solids; remaining material collected and dried at 105 °C for 24h, and dried sample weighed. Results expressed as dry weight per litre.
CSS150	Coarse suspended solids measurement. 0.25 L sample of mixed liquors, diluted to 2.5 L, stirred at 200 rpm for 15 min, passed through 150µm sieve for 2 minutes with gentle rinsing using tap water to assist sieving and to isolate filter solids, remaining material collected and dried at 105 °C for 24h, and dried sample weighed. Results expressed as dry weight per litre.
CSS500	Coarse suspended solids measurement. 0.5 L sample, diluted to 1 L, stirred manually for 1 min, passed through 500 µm mesh sieve for 2 minutes with gentle rinsing using tap water to assist sieving and to isolate filter solids, remaining material collected and dried at 105 °C for 24h, and dried sample weighed. Results expressed as dry weight per litre.
CSS4000	Very coarse and reconstituted suspended solids measurement. Net, ~4mm diameter 2D hexagonal mesh, used to sieve ~1 m^3 of mixed liquor in the MBR tank by passing the net through region above membrane units ten times. Net lifted above liquid level after each pass to allow excess liquid to drain off. Retained material gently rinsed with water and weighed. Results expressed as weight of wet solids per litre.
CSSR(a-d)	Coarse solids reconstitution test developed by the researchers. Sample of solids captured by the CSS4000 method (a) Captured material manually defragmented, dewatered and rolled for 1 minute before assessing mechanical integrity through suspending one end of the agglomerated solids. (b) Solids washed with water and mechanical integrity test repeated. (c) 50% of washed solids soaked for 24h in detergent and mechanical integrity test repeated. (d) Remaining 50% of washed solids soaked for 24h in 5% sodium hypochlorite and mechanical integrity test repeated.

Table 2: Coefficient of variation values, three test repetitions

Test name	*Pore size*	*Coefficient of variation*	*Main experiment no. of repetitions*
Filterability	2.5µm	22%	3
CSS-A	150µm	34%	2 (CSS-B)
CSS150	150µm	4.0%	2
CSS500	500µm	9.9%	1
CSS4000	4mm	26%	3

Table 3: Results for one sided T-test with Kruskal-Wallis analysis for macerating and non-macerating screenings handling plants.

Groups:		*nMSH plants, n = 4*		*MSH plants, n = 6*		*p value*	
Parameter	*Unit*	*Mean*	*Std error*	*Mean*	*Std error*	*T-test*	*KW-test*
MLSS	mg/l	13,954	3,009	16,059	2,450	0.15	0.29
Filterability	ml/5min	12.2	0.84	6.47	1.25	0.002	0.01
CSS-B	mg/l	741	159	918	122	0.21	0.52
CSS150	mg/l	567	107	850	102	0.05	0.06
CSS500	mg/l	196	37.6	326	46	0.03	0.03
CSS4000	mg/l	53.3	18.3	250	76.5	0.03	0.02

Table 4: Results for one sided T- test with Kruskal-Wallis analysis for clogging resistant and susceptible plants.

Groups:		*Clogging-resistant plants, n = 5*		*Clogging-susceptible plants, n = 5*		*p value*	
Parameter	*Unit*	*Mean*	*Std error*	*Mean*	*Std error*	*T-test*	*KW-test*
MLSS	mg/l	14,021	2,610	16,413	2,562	0.09	0.25
Filterability	ml/5min	11.2	1.13	6.27	1.52	0.02	0.03
CSS-B	mg/l	725	124	969	135	0.11	0.25
CSS150	mg/l	572	82	902	107	0.02	0.03
CSS500	mg/l	207	30	341	53	0.04	0.01
CSS4000	mg/l	75.3	26	267	91	0.05	0.05

Table 5: Relationships observed between MLSS, Filterability and CSS tests data.

Parameters:	*MLSS*		*Filterability*	
	Relationship	R^2	Relationship	R^2
Filterability	Logarithmic	0.008	-	-
CSS-B	Linear	0.09	Linear	0.07
CSS150	Linear	0.14	Linear	0.27
CSS500	Logarithmic	0.06	Exponential	0.77
CSS4000	Logarithmic	0.06	Exponential	0.94